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Fatigue crack growth in alumina particulate reinforced Ni3Al alloy composites, IC-50/Al2O3, fabricated using a spray deposition and co-injection process was examined and compared with that in a conventional nickel-based superalloy, Inconel 718. Single edge-notched fatigue crack growth specimens were tested under constant $\Delta K$ conditions using a custom fully automated servo-hydraulic test system. The results indicate that the threshold value of $\Delta K$ is at least as high as that for monolithic IC-50, regardless of whether the composite is in the as-sprayed or HIPed condition. However, the HIPed microstructure exhibits a higher fatigue crack growth resistance in the Paris law regime compared to the as-sprayed material. Both composites exhibit a lower crack growth resistance compared to that for unreinforced IC-50. Compared with Inconel 718, the present IC-50 MMCs consistently exhibit substantially better fatigue crack growth resistance properties.			
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## **FATIGUE MECHANISMS IN METALLIC MATRIX COMPOSITES**

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## **Objectives**

The objective of the present research is to develop a fundamental understanding of the intrinsic relationships between solidification processing and the development of fatigue damage in metallic matrix composites (MMCs) under cyclic loading conditions. Specifically, the overall goal of the augmentation is to understand of the influence of spray atomized and co-injected microstructures on the growth of fatigue cracks in particulate reinforced metallic matrix composites. This understanding is then used to develop the optimum processing route for superior fatigue resistance and endurance.

## **Status of Effort**

This four-year augmentation was initiated on July 1, 1992; the present report covers the final year from July 1, 1995 - June 31, 1996. Fatigue crack growth in alumina particulate reinforced Ni<sub>3</sub>Al alloy composites, IC-50/Al<sub>2</sub>O<sub>3</sub>, fabricated using a spray deposition and co-injection process [1-3], was examined and compared with that in a conventional nickel-base superalloy, Inconel 718. Single edge-notched (SEN) fatigue crack growth specimens were tested under constant  $\Delta K$  conditions using a custom fully automated servo-hydraulic test system. The results indicate that the threshold value of  $\Delta K$  is at least as high as that for monolithic IC-50, regardless of whether the composite is in the as-sprayed or HIPed condition. However, the HIPed microstructure exhibits a higher fatigue crack growth resistance in the Paris law regime compared to the as-sprayed material. Both composites exhibit a lower crack growth resistance compared to that for unreinforced IC-50 [4]. Compared with Inconel 718, the present IC-50 MMCs consistently exhibit substantially better fatigue crack growth resistance properties.

We also compared the average microcrack growth rate in unnotched cylindrical samples with the crack growth rate for single edge notched specimens of Inconel 718. The growth of the cracks was monitored by periodically interrupting the fatigue experiments and performing an acetate replication of the entire gage section [5]. A deceleration observed for the microcrack growth in Inconel 718. Comparison of the value of  $\Delta J$  for this transition with the threshold value of  $\Delta J$  indicated by the SEN crack growth data indicates that the observed deceleration is not the result of the onset crack closure effects. Rather, it is shown to correspond to a transition between the growth of microcracks that are generally within a single grain to the growth of microcracks through multiple grains [6, 7].

## Accomplishments/New Findings

### 1. Research Highlights

We have studied the mechanisms that control fatigue crack growth in spray atomized and co-injected  $\text{Ni}_3\text{Al}$  (IC-50) matrix composites by conducting automated crack growth experiments under controlled  $\Delta K$  conditions. During these tests the value of  $\Delta K$  is held constant for a sufficiently long period to accurately measure crack growth rate and then successively reduced to lower  $\Delta K$  levels until the threshold  $\Delta K$  value is indicated. The fatigue crack growth rates for the IC-50/ $\text{Al}_2\text{O}_3$  IMCs, compared with Inconel 718 and monolithic IC-50, are plotted versus  $\Delta K$  in Figure 1. Among the present MMCs, a hot isostatically pressed (HIPed) IC-50/ $\text{Al}_2\text{O}_3$  IMC exhibits a greater fatigue crack growth resistance. This may be attributed to the reduced amount of porosity in the HIPed IC-50 MMC compared with that in the as-sprayed composite. Another possible enhancement is that the HIPing process enables boron atoms to diffuse to the grain boundaries, increasing their cohesive strength. We note, however, that the values of  $\Delta K_{th}$  are essentially the same regardless of whether the composite is in the as-sprayed or HIPed condition.

We also found that the fatigue crack growth resistance for monolithic IC-50 and HIPed IC-50/ $\text{Al}_2\text{O}_3$  MMCs are substantially better than that for Inconel 718 with either a solution treated double annealed (STDA) or solution treated water cooled (STWC) heat treatment. The crack growth behaviors of the specimens, machined either vertically or horizontally within the spray deposit, shows no difference. This finding is consistent with metallographic evidence that the microstructure of this material is effectively isotropic in nature.

Small fatigue crack growth in unnotched specimens of STWC Inconel 718 under constant strain amplitude conditions were also studied and are compared with SEN fatigue crack growth data. The surface state of the LCF damage [5] was characterized with two parameters: average surface crack length and surface crack density. These parameters were measured by periodically interrupting the experiments and producing acetate replicas of the entire gage section. An in-situ laser scanning technique was also applied to monitor the surface state of the LCF specimens. The results from this technique were found to correlate well with the microcrack density measurements [5].

Crack growth rate data for two Inconel 718 LCF samples,  $\Delta\epsilon_a = 0.6\%$  and  $\Delta\epsilon_a = 0.8\%$ , are plotted with SEN data as a function of  $J$ -integral range,  $\Delta J$ , in Figure 2. The

details of  $\Delta J$  calculation and estimation of Young's modulus,  $E_{cyclic}$ , are reported elsewhere [6-8]. We note that the small crack growth for the LCF specimens exhibit a short-lived crack growth deceleration for a value of  $\Delta J$  that is about one order magnitude less than  $\Delta J_{th}$  as indicated from the SEN experiments. Hence, it appears unlikely that the onset of crack closure effects could be the cause of the observed deceleration.

The observed deceleration in microcrack growth rate occurs in the LCF specimens when the average surface crack length is about 20  $\mu\text{m}$ . Comparison of this value with the average grain diameter of 27  $\mu\text{m}$  suggests that the deceleration results from a transition from crack growth within a single grain to that through multiple grains. We note that it is likely that the resolved shear stress for slip in the subsequent grains the microcracks growth through are not as high as that for the grain in which it initiates. In this case, the crack tip deformation that facilitates crack should initially become more difficult as the crack front passes from a single grain into multiple grains. Thus, it is reasonable that the observed transition results in a temporary reduction in crack growth rate even though the value of  $\Delta J$  continually increases as the crack grows.

The present laser scanning systems outputs a parameter called the defect frequency which corresponds to the number of grains at the surface which contain microcracks. Both mean defect frequency and microcrack density are plotted against strain amplitude in Figure 3 for the point in the LCF experiments where the microcrack density reaches a maximum (saturation). It can be seen in this figure that the overall microcrack density increases linearly with strain amplitude. This trend is reasonable since the saturation microcrack density should directly depend on the density of persistent slip bands at the surface which has been shown to be linearly dependent on the plastic strain amplitude [9]. The relationship between strain amplitude versus mean defect frequency is also approximately linear in Figure 3, particularly below a strain amplitude of 0.8%. It is likely that the deviation from linearity above this strain amplitude is due to fact that a significant number of grains containing multiple cracks are present. For these grains, the multiple cracks are counted as a single crack since their spacing is less than the laser beam diameter. As a result, the defect frequency is lower than what would be expected from the absolute increase in microcrack density with strain amplitude.

Number of cycles to the saturation of the defect frequency,  $N_s$ , plotted against the number of cycles to failure,  $N_f$ , for the present Inconel 718 specimens is illustrated in Figure 4. This plot indicates a linear relationship between  $N_s$  and  $N_f$  with a slope that is approximately equal to 1/5. This relationship indicates that low cycle fatigue life can be

predicted reasonably well from the onset of the maximum mean defect frequency level for the present material. Taking this together with the results shown in Figure 3 further suggests that the number of cycles to reach the maximum microcrack density is linearly related to the number of cycles to failure.

## 2. Significance to the Field

The significance of the present research is based on the fact that the nature of the mechanisms which control the development of fatigue damage in intermetallic matrix composites is not well understood. Most detailed studies of fatigue mechanisms have been conducted with monolithic metal and intermetallic alloys. The present results demonstrate that the fatigue crack growth resistance of a novel spray atomized and co-deposited  $\text{Ni}_3\text{Al}/\text{Al}_2\text{O}_3$  MMC is substantially better than that of a nickel-base superalloy, Inconel 718. It is also apparent that the crack growth resistance in the present composite is essentially isotropic from the similarity of the data in Figure 1 for the horizontally and vertically oriented specimens. A laser scanning technique has been further developed to monitor the formation and growth of fatigue microcracks in unnotched specimens. The results from this study indicate that the number of cycles to reach a maximum in the microcrack density is linearly related to the fatigue life of the specimens. This work has enabled us to establish an understanding of the influence of microstructure on the fatigue damage resistance of novel intermetallic matrix composites. It is anticipated that this research will lead to the development of other novel metallic matrix composites with superior mechanical properties under cyclic as well as static loading conditions.

## 3. Relevance to the Air Force's Mission and Potential Applications

Future engine designs for military aircraft will require new high temperature materials that exhibit prolonged lifetimes under fatigue conditions. Spray atomization of  $\text{Ni}_3\text{Al}$  has been shown in the past to improve room temperature ductility, however this benefit often comes with the drawback of significant porosity (>6 vol.%) in the deposited material. The present  $\text{Ni}_3\text{Al}/\text{Al}_2\text{O}_3$  IMC has a relatively fine grain size and a very low porosity level (<1 vol.%). This combination facilitates higher room temperature ductility as well as improved fatigue crack growth resistance. Such a development makes this composite potentially useful in hot section components of military and commercial jet turbine engines. Blades, vanes and stators could potentially operate at higher temperatures and experience longer lifetimes with the excellent oxidation resistance of  $\text{Ni}_3\text{Al}$ . The introduction of alumina particulates that are less than 3  $\mu\text{m}$  in diameter also appears to have a beneficial effect of improving the fatigue resistance by reducing interfacial and particulate

cracking. These improvements ultimately allow the design specifications to incorporate a longer service life. The increased fatigue damage resistance of the present MMCs can reduce costs in two ways: (1) lighter structures may be developed which increase fuel efficiency and (2) maintenance and component replacement operations are less frequent. In addition, the fatigue data obtained in developing the MMCs under investigation are required for accurate predictions of the fatigue failure of future MMC components. The results of the present research program provide a better understanding of the nature of fatigue crack growth in recently developed intermetallic matrix composites that are potentially useful in aerospace applications. This understanding will assist in the design of advanced materials with a greater resistance to failure to meet the design criteria anticipated for both future civilian and military aerospace vehicles.

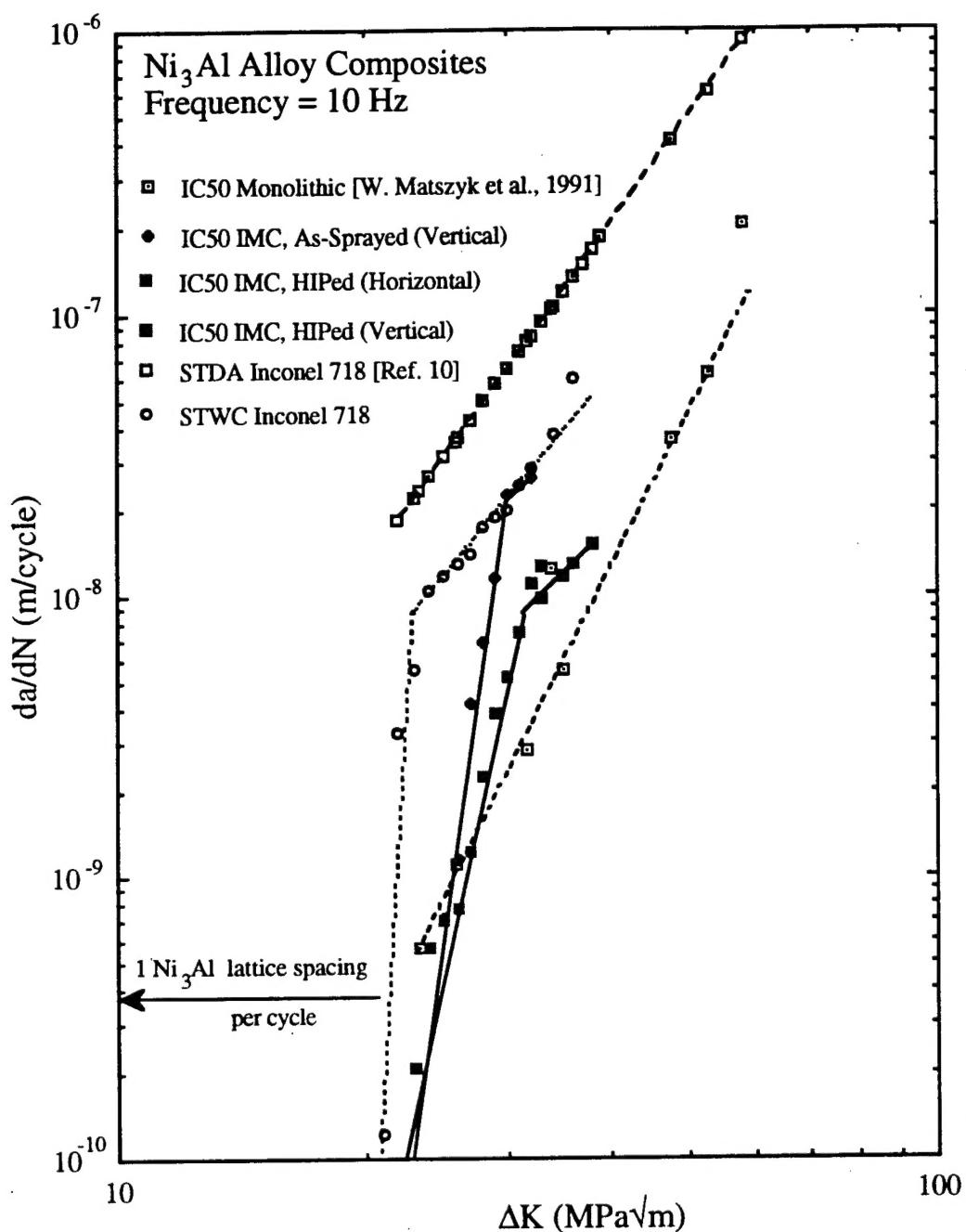


Figure 1. Fatigue crack growth rate versus  $\Delta K$  for the present IC-50/Al<sub>2</sub>O<sub>3</sub> and STWC Inconel 718 compared with that for monolithic IC-50 and STDA Inconel 718 respectively.

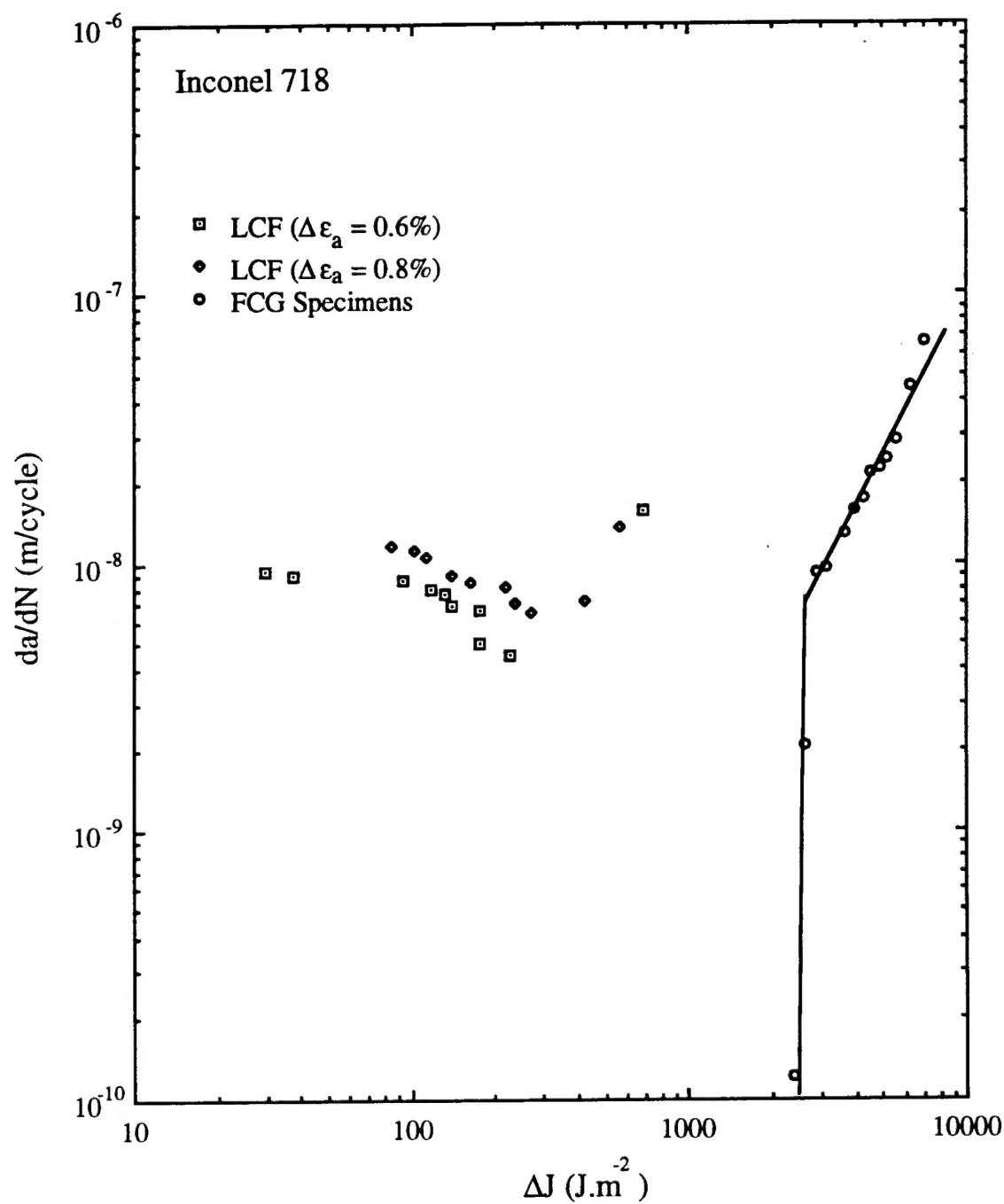


Figure 2. Fatigue crack growth rate versus  $\Delta J$  for the present SEN specimens of STWC Inconel 718 compared with corresponding LCF data for surface microcracks.

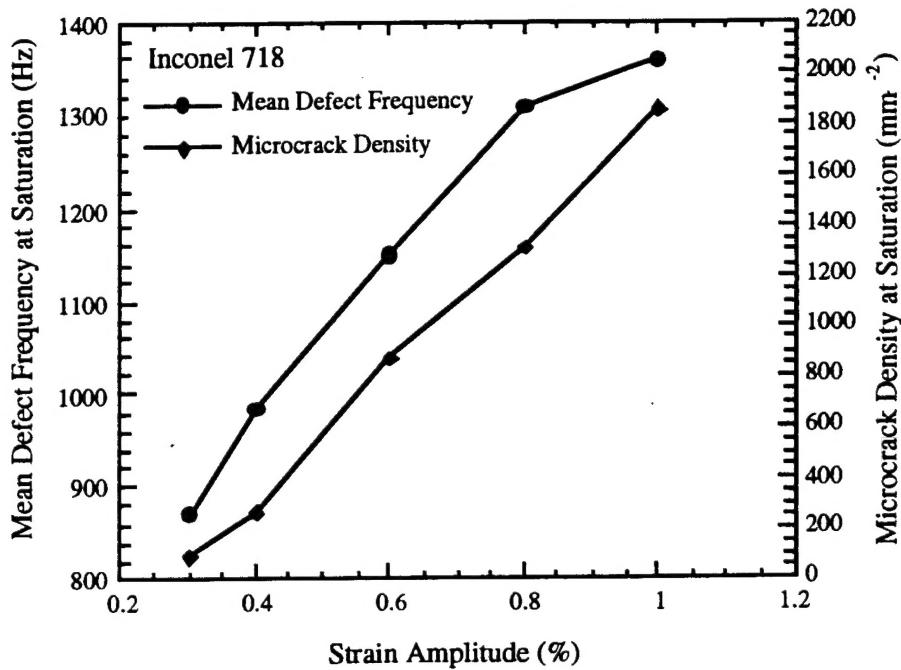


Figure 3. Both mean microcrack density and mean defect frequency versus strain amplitude at the point in the tests when the mean surface microcrack density reaches a maximum (saturation).

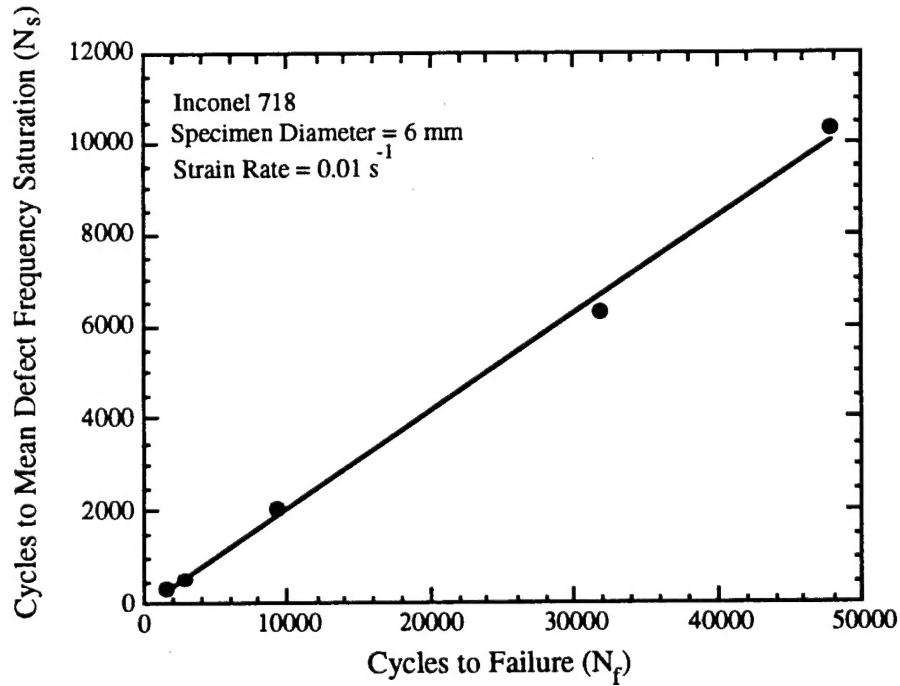


Figure 4. Data plot exhibiting the linear relationship between the cycles to failure and the cycles to reach the observed saturation in mean defect frequency.

### **References**

1. X. Liang, H.K. Kim, J.C. Earthman, and E.J. Lavernia, "Microstructure and Elevated Temperature Behavior of a Spray-Atomized and Co-Deposited  $\text{Ni}_3\text{Al}/\text{SiC}/\text{TiB}_2$  Intermetallic Matrix Composite," Materials Science and Engineering, Vol. A153 (1992), 646-653.
2. X. Liang and E.J. Lavernia, "Interfacial Behavior in a  $\text{Ni}_3\text{Al}/\text{TiB}_2$  Intermetallic Matrix Composite," Materials Science and Engineering, Vol. A153 (1992), 654-661.
3. X. Liang and E.J. Lavernia, "Solidification and Microstructure Evolution during Spray Atomization and Deposition of  $\text{Ni}_3\text{Al}$ ," Materials Science and Engineering, Vol. A161 (1993), 221-235.
4. W. Matuszyk, G. Camus, D.J. Duquette, and N.S. Stoloff, "Effects of Temperature and Environment on the Tensile and Fatigue Crack Growth Behavior of a  $\text{Ni}_3\text{Al}$ -Base Alloy," Metallurgical Transactions A-Physical Metallurgy, Vol. 21A, 2967-2976 (1990).
5. K.J.C. Chou and J.C. Earthman, "Characterization of Low Cycle Fatigue Damage in Inconel 718 by Laser Light Scanning," Journal of Materials Research, submitted for publication.
6. J.C. Earthman, "Characterization of Small Crack Growth in 12% CrMoV steel Under High Temperature, Low Cycle Fatigue Conditions," Materials Science and Engineering, Vol. A132, 89-95 (1991).
7. D. Fournier and A. Pineau, "Low Cycle Fatigue Behavior of Inconel 718 at 298 K and 823 K," Metallurgical Transactions A-Physical Metallurgy, Vol. 8A, 1095-1105 (1977).
8. K.B. S. Rao, S. Kalluri, G.R. Halford and M.A. McGaw, "Serrated Flow and Deformation Substructure at Room Temperature in Inconel 718 Superalloy During Strain Controlled Fatigue," Scripta Metallurgica et Materialia, Vol. 32, No. 4, 493-498 (1995).
9. A.T. Winter, Philosophical Magazine, Vol. 30, 719 (1974).
10. James E. Campbell, Metals Handbook Ninth Edition, American Society for Metals, Metals Park, Ohio, Vol. 3, 753 (1980).

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1. D. E. Lawrynowicz and E. J. Lavernia, "A Review of Sensors and Techniques to Monitor Process Parameters during Spray Atomization and Deposition," *Journal of Materials Science*, Vol. 30, pp. 1125-1138, 1995.
2. D. Lawrynowicz, B. Li, and E. J. Lavernia, "Particle Penetration Mechanisms of a Spray Atomized and Co-Deposited Ni<sub>3</sub>Al+B/Al<sub>2</sub>O<sub>3</sub> Metal Matrix Composite," submitted to *Metallurgical Transactions*, 1996.
3. K. J. C. Chou and J. C. Earthman, "Characterization of Low-Cycle Fatigue Damage in Inconel 718 by Laser light Scanning," submitted to *Journal of Materials Research*, 1996.

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1. K. J. C. Chou and J. C. Earthman, "Characterization of Low-Cycle Fatigue Damage in Inconel 718 by Laser Light Scanning ", TMS International Conference on High Temperature Materials Characterization, Feb. 13-17, Las Vegas, Nevada, 1995.
2. K. J. C. Chou and J. C. Earthman, "Characterization of Low-Cycle Fatigue Damage in Inconel 718 by Scattered Laser light Scanning," in *Nondestructive Evaluation (NDE) and Materials Properties (III)*, ASM/TMS Materials Week '96, Cincinnati,, OH, 1996.

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(provided the Ni<sub>3</sub>Al alloy used to process the material discussed in the present report)